High Entropy Heusler Alloys

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Examined by:

Seal of the School

DECLARATION BY STUDENT

I hereby declare that the data presented in this Dissertation report entitled. **“High Entropy Heusler Alloys”** is based on the results of investigations carried out by me in the Physics Discipline at the School of Physical and Applied Sciences. Goa University under the Supervision of Dr. Elaine Dias and the same has not been submitted elsewhere for the award of a degree or diploma by me. Further. I understand that Goa University or its authorities will be not be responsible for the correctness of observations / experimental or other findings given the dissertation.I hereby authorize the University authorities to upload this dissertation on the dissertation repository or anywhere else as the UGC regulations demand and make it available to any one as needed.

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Place: Goa University

COMPLETION CERTIFICATE

This is to certify that the dissertation "High Entropy Heusler Alloys" is a bonafide work carried out by Ms. Rajvi Naik under my supervision in partial fulfilment of the requirements for the award of the degree of M.Sc in Physics at the School of Physical and Applied Sciences, Goa University.

Supervisor: Dr. Elaine Dias

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Abstract

In my project, we have investigated the strategy of adding high entropy in Ni_2MnGa which is a Heusler alloys. Initially, we laid the foundation for our project by preparing normal Ni_2MnGa and characterized it which was verified by the results according to the literature, this was the First objective of my project. We later introduced high entropy in Mn site. we accomplished this by substituting five additional elements for Mn such that the e/a ratio of these elements equals the number of valence electrons in Mn. This was made possible by the discovery that the e/a ratio of V Cr Mn Fe and Co is seven, which is equal to the valence electron of Mn, hence High entropy was introduced by selecting this alloys. which was the second objective of my project.Both these alloys were prepared by annealing them at 750 ^oC And lastly we characterized these high entropy heusler alloys by various characterization techniques which include Room Temperature powder $X - Ray$ Diffraction this was done to verify the structural studies, Temperature dependent resistivity which was done using four-probe method this technique is used to characterizing the electrical properties of alloys and lastly Differential Scanning Calorimetry characterization was done, this is done to check transition temperature

Contents

Chapter 1

Introduction

1.1 Historical background

Fritz Heusler was a German chemist and mining engineer who founded Heusler Alloys in 1901. Everything began with a small stroke of luck, while working at the German smelter Isabellenhütte, Heusler experimented with various metal combinations. He produced an alloy that included roughly equal amounts of copper (Cu), manganese (Mn), tin (Sn), aluminum (Al), arsenic (As), antimony (Sb), and bismuth (Bi).Remarkably, this fairly common alloy had an unexpected characteristic, it was ferromagnetic. The fact that the three elements Cu, Mn, and the third element are not intrinsically magnetic in their pure forms made this discovery noteworthy.The scientific community was rocked when a magnetic alloy made of non-magnetic elements was discovered. It questioned the then current comprehension of magnetism. After the first discovery, more research revealed the mysteries of Heusler alloys.[8] It was found by researchers that Heusler alloys had a distinct crystal structure that has never been seen before. This structure, which is now referred to as the Heusler structure, is distinguished by a particular atom arrangement in a face-centered cubic lattice with extra ordering.The apparent magnetism was determined to be caused by the special arrangement of atoms in the Heusler structure. It was discovered that a key factor in producing magnetic characteristics was the arrangement of various components inside the lattice and how they were combined.[8]Heusler alloys were first discovered, which spurred additional investigation and led to the study of a larger spectrum of compositions. There has been a growing body of research on the many functional qualities that Heusler alloys display. Among them are:

- Shape Memory Effect: When a certain stimulus, such as heat or a magnetic field, is applied, some Heusler alloys have the ability to regain their original shape after deformation.
- Giant Magnetostriction: These alloys are useful for actuators and micro-positioning devices because they can change significantly in shape when subjected to a magnetic field.
- Spintronics: Heusler alloys' ability to manipulate electron spin suggests new uses for information storage and gadgets.
- Thermoelectrics: Heusler alloys have the potential to be used in energy conversion applications since they can turn heat into electricity or vice versa.

1.2 Heusler alloys

Heusler alloys are ternary intermetallic compounds with the composition XYZ and crystallize in the $L2_1$ structure . Heusler alloys can generally classified as full Heusler with formula X_2YZ and half Heusler with formula XYZ. As shown in the figure 1.1, the elements which are highlighted in orange colour can occupy the X position whereas the elements which are highlighted in blue can occupy the Y site and green colour can occupy the Z site. The $L2_1$ structure is a cubic structure, this cubic structure depicts that the X atoms is occupying the (0,0,0) and ($\frac{1}{2}$, $\frac{1}{2}$,$\frac{1}{2}$), Y atoms is occupying the ($\frac{1}{4}$, $\frac{1}{4}$, $\frac{1}{4}$) and Z atoms is occupying the ($\frac{3}{4}$, $\frac{3}{4}$, $\frac{3}{4}$) positions. Ni_2MnGa is a well studied example of Heusler alloy which are known for its magnetic shape memory and $L2_1$ structure

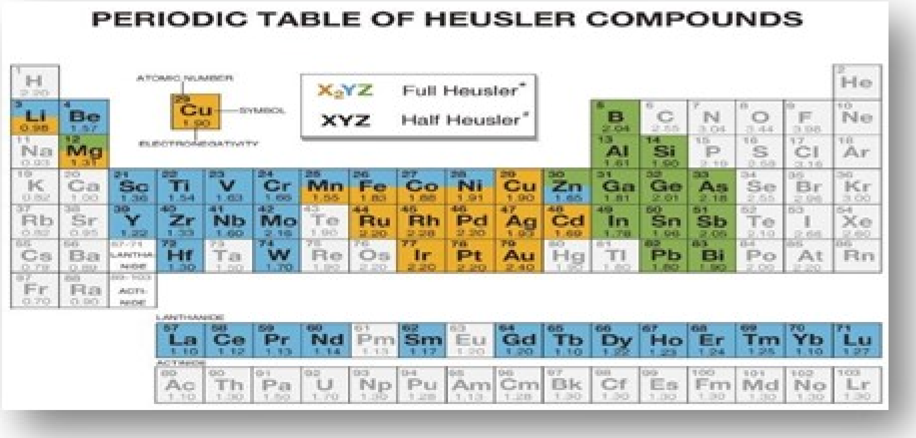


Figure 1.1: periodic table of heusler compound

1.3 Shape Memory Alloy

Shape Memory Alloys (SMAs), are a class of metallic materials that are known to remember their shape and have the ability get back to its shape when it subjected to the appropriate heat procedure. Generally, at relatively low temperature, these materials can plastically be deformed, and when exposed to some higher temperature will return back to their shape prior to the deformation. Some materials exhibit shape memory only upon heating also known as one-way shape memory and some materials undergo a change in shape on recooling known as two-way shape memory. In the case where the shape memory alloys are magnetic, they exhibit magnetic shape memory effect (MSM). This effect was first discovered in Heusler alloys. In its stoichiometric composition, ferromagnetic Ni_2MnGa undergoes a martensitic transformation from $L2_1$ cubic structure to an orthorhombic structure

1.4 Ni_2MnGa

Ni_2MnGa belongs to the group of 'shape memory effect' with a thermoelastic phase change at 200 K, transforming from a cubic $L2_1$ Heusler structure to a phase of lower symmetry.[9] When plastically deformed in the low temperature martensitic phase and external load removed, the material regains its original shape when heated above the transition temperature. This transformation is described as a simple contraction along one of the $< 100 >$ directions of the cubic cell,[4] without any change in atomic positions[1]. Despite the large deformation, the phase transition is reversible, allowing a single crystal to cycle through it many times without breaking. Shape memory alloys can produce displacement, force, or a combination of the two as a function of temperature, helping to solve various problems in engineering and medicine[5] On cooling, stoichiometric Ni_2MnGa undergoes a martensitic phase transition at 200 K, with powder neutron diffraction measurements showing a cubic Heusler $L2_1$ structure above T_M and a tetragonal structure below it. The transformation process in Ni_2MnGa depends on the internal strain in specimen.

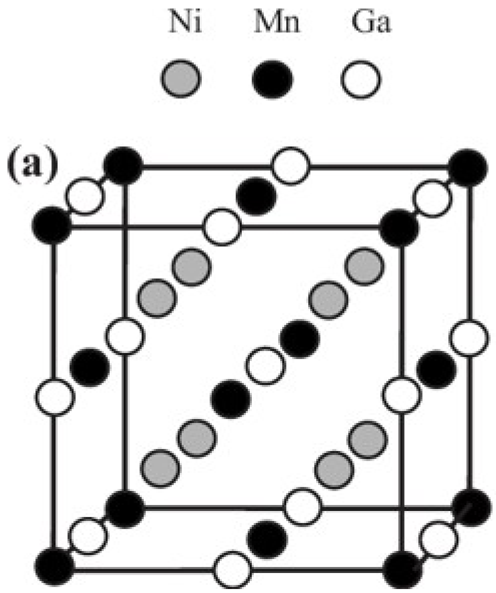


Figure 1.2: Ni_2MnGa

1.5 High Entropy Alloys

The definition of HEAs has been a subject of discussion; however, two versions have gained widespread acceptance. On the basis of the compositional requirements, the initial definition was put forth in 2004. An alloy with at least five primary metal elements and an atomic percentage of 5–35 % for each is referred to as high-entropy. Alloys with little or no atomic percentage of elemental additives less than 5 % are implicitly included in this definition. This definition can be expressed as follows:

$$\begin{aligned} n_{major} \geq 5, \quad 5\,\text{at.\%} \leq c_i \leq 35\,\text{at.\%} \\ n_{minor} \geq 0, \; c_j \leq 5\,\text{at.\%} \end{aligned}$$

where n_{major} and n_{minor} are the number of primary and secondary elements, respectively. c_i and c_j are the atomic percentages of primary and secondary el-

$$\Delta S_{mix} \geq 1.61R$$

ements, respectively. As per this definition, the high-entropy alloy system can consist of many non-equimolar elements, multiple minor elements, or an equimolar ratio. The utilization of high mixing entropy to promote the formation of the solid solution phase is the fundamental idea behind this concept. Entropy is the foundation of the second definition. According to this definition, an alloy is considered HEA if its configurational entropy in its random solution state is more than 1.61R, where R is the gas constant. One way to state this definition is Entropy is a fundamental state function and a quantity that describes chaos from a thermodynamic perspective. The higher the level of confusion, the higher the system's entropy. Boltzmann's thermodynamic statistical principle states the entropy of a system can be expressed as

$$S = k \log W \tag{1.1}$$

where,

k is Boltzmann constant, related to the molar gas constant R ($R = 1.381013 J/K$)

W is the thermodynamic probability,

which represents the total number of microscopic states contained in the macroscopic state

Configurational entropy, hybrid entropy, or mixed entropy is the term used to describe the rise in system entropy induced by the various atom configurations of different alloying elements when the heat of mixing is not taken into consideration. The atomic arrangement has the largest effect on entropy for multi-principal HEAs. The configurational entropy of the atomic arrangement dominates the system's entropy if the influence of other configurations on the entropy is ignored, which is acceptable.[9]

1.6 Literature Review

A great deal of research has been done to comprehend magnetic shape memory phenomena and, specifically, to find better alloys that exhibit high strains, noticeable thrust, and quick reaction times. One of the "shape memory effect" alloys that is generating a lot of interest right now is Ni_2MnGa. This phenomenon in Ni_2MnGa originates from the thermoelastic phase transition from the cubic $L2_1$ Heusler structure to a phase of lesser symmetry that occurs at T_M upon cooling by 200 K.[10] When heated above the transition temperature, the material that was plastically deformed in the low-temperature martensitic phase and the external load removed will return to its original shape. The transformation has been explained as a straightforward contraction along one of the $< 100 >$ directions of the cubic cell, with no change in the atomic locations, based on early neutron diffraction data At high temperatures,The cell deforms strongly (c/a = 0.94), yet the cell volume decreases by only 1%. This phase transition is unique in that a single crystal can cycle through it repeatedly without breaking and is reversible despite the significant distortion. Depending on the temperature, the alloys can create a displacement, a force, or a combination of the two while regaining their shape. Shape memory alloys are helping to tackle a wide range of problems in both engineering and medicine because of these unique and amazing qualities. Ni_2MnGa exhibits a nonmagnetic $L2_1$ Heusler structure with a lattice constant of a = 5.825 $\AA$.[6] At temperatures T_c 380 K, Ni_2MnGa experiences a magnetic phase shift from a paramagnetic to a ferromagnetic state. In the $Fmmm$ space group, a martensitic phase transformation takes place at temperature T_M= 276 K, resulting in a distorted tetragonal lattice with a = 5.92 $\AA$ and c = 5.54 $\AA$ (c/a = 0.94).[10] Since magnetic shape memory alloys have been extensively investigated for various applications in the past decade,however there is no proper understanding of introducing high entropy in heusler alloys thereforethe main aim of my project is as follows

(i) **To prepare and characterise Ni_2MnGa Heusler alloy**

(ii) **To introduce high entropy at Mn site in the formula Ni_2MnZ where Z = Ga and by synthesising $Ni_2V_{0.2}Cr_{0.2}Mn_{0.2}Fe_{0.2}Co_{0.2}Z$.**

(iii) **To characterise the high entropy Ni_2MnGa Heusler alloys.**

Chapter 2

Experimental techniques

2.1 Sample calculation

According to sample stoichoimetry, elements weight are calculated to form 1g of sample. Later this 1g of sample is modified in order to get 1.5g of sample.

Ni	Mn	Ga	V	Cr	Fe	Co
58.71	54.938	69.72	50.942	51.996	55.847	58.933

Table 2.1: Elements with atomic weights

Weights required for Ni_2MnGa

Ni_2MnGa		
Ni	Mn	Ga
0.7276 g	0.3404 g	0.4320 g

(i) Ni_2MnGa

$$= (2 \cdot 58.71) + 54.938 + 69.72 \tag{2.1}$$

$$= 242.078g \tag{2.2}$$

$$\tag{2.3}$$

- Amount of Ni required

$$= \frac{2 \cdot 58.71}{242.078} \tag{2.4}$$

$$= 0.4851g \tag{2.5}$$

- Amount of Mn required

$$= \frac{54.938}{242.078} \tag{2.6}$$

$$= 0.2269g \tag{2.7}$$

- Amount of Ga required

$$= \frac{69.72}{242.078} \tag{2.8}$$

$$= 0.2880g \tag{2.9}$$

These are the weights required to prepare 1g of sample. In order to make

1.5g of sample we should multiply these weights with 1.5

For 1.5g of Ni

$$= 1.5 \cdot 0.4851 \tag{2.10}$$

$$= 0.7276g \tag{2.11}$$

For 1.5g of Mn

$$= 1.5 \cdot 0.2269 \tag{2.12}$$

$$= 0.3404g \tag{2.13}$$

For 1.5g of Ga

$$= 1.5 \cdot 0.2880 \tag{2.14}$$

$$= 0.4320g \tag{2.15}$$

Similarily calculations were done for the high entropy sample and the following table is about thr required weights required for 1.5g of sample. Weights required for high entropy Ni_2MnGa

HE Ni_2MnGa						
Ni	V	Cr	Mn	Fe	Co	Ga
0.7290g	0.063g	0.0650g	0.0690g	0.0690g	0.0740g	0.4327g

2.2 Sample Preparation

A 1.5 g sample of Ni_2MnGa was prepared by repeated melting of the constituent Nickle, Manganese Gallium elements in stoichiometric proportions in an argon arc furnace. First Nickle powder used to synthesise the sample was characterised using XRD technique to check for purity. Next Manganese was weighed and cleaned using metal cleaner. For this manganese pieces were placed in a beaker containing the metal cleaner in a ultra sonicator for 15 minutes. The piece was then washed with Iso propyl alcohol the process was repeated several times till the shiny surface was seen . Next Gallium was measured according to its stoichiometric weight as shown in the table.

Ni_2MnGa:

Ni	Mn	Ga
0.7276	0.3404	0.4320

Table 2.2: stoichiometric weights

Later high entropy was introduce in Mn site and $Ni_2V_{0.2}Cr_{0.2}Mn_{0.2}Fe_{0.2}Co_{0.2}Z$ (Z=Ga) compound were prepared, A 1.5g sample of $Ni_2VCrMnFeCoGa$ was prepared in the similar way. The following table is about the weights required fore preparing

$Ni_2VCrMnFeCo$ Z (Z=Ga):

Ni	V	Cr	Mn	Fe	Co	Z
0.7288	0.0632	0.0645	0.0682	0.0693	0.0732	0.4327

Table 2.3: stoichiometric weights

The powder and solid beads were sealed under a reduced argon atmosphere in a quartz ampoule and annealed at 750∘C after which they were quenched in ice cold water

2.2.1 Set up used

High entropy was introduced in Mn site and $Ni_2V_{0.2}Cr_{0.2}Mn_{0.2}Fe_{0.2}Co_{0.2}Z$ (Z=Ga) compound were prepared. To prepare the entire compound first Nickel bead was formed, then Mn was prepared but replacing it with 5 elements which are VCrMnFeCo. This preparation was done by vacuum arc melting process which was flushed with argon atmosphere. Lastly all the components i.e. the nickel bead, the HE Mn bead and the Z (Z= Ga) components were put together and preparation was done by vacuum arc melting process which was flushed with argon atmosphere.

Arc Melting

Arc Melting is basically used for melting metals, mainly to form alloys. consists of 3 main parts which is the power source (TIG-600amp), chiller and the vacuum unit. The vacuum unit with rotary and diffusion pump has a vacuum of 10-3 bar. Heating is done via an electric arc struck between a tungsten electrode and metals placed in a crucible in the copper chamber. The chiller cools both the copper hearth and the tungsten electrode by circulating cold water. In vacuum arc melting the chamber is evacuated and then it is back filled with argon gas. Therefore, melting is performed in argon atmosphere, the electric arc generates a huge amount of heat concentrated near the sample, which melts the metal together into an ingot. The following images are of arc furnace used during the experiment

Figure 2.1: Arc Furnace

Figure 2.2: Arc Furnace with eye shield

(i) Making High Entropy Ni_2MnGa

In order to make High Entropy Ni_2MnGa with the help of this process, this process was divided in 3 parts. First the nickle bead was prepared after which high entropy manganese was made and in the end all elements were placed.

(a) Nickle bead formation

To prepare the Nickel bead first Nickle powder taken little in excess. The following table is about the weight taken before the process

Ni taken	Ni required
0.8116g	0.7288g

Table 2.4: weights

Three cycles were done of vacuum and argon, After 3 cycles the nickel powder is melted into a bead. It is melted at 75 amperes current, and is flipped 3 times this was done to ensure homogeneous composition

(b) High Entropy Manganese formation

Manganese was substituted with 5 other metals and placed according to vapour pressure (Co, Cr, Fe, Mn, V). Three cycles were done of vacuum and argon, kept at different time.

After 3 cycles the elements are melted into a bead. It is melted at 75 amperes current, and is flipped 3 times

(c) Hight entropy Ni_2MnGa bead formation

The nickel bead, HE Mn and gallium was placed in the copper earth. Three cycles were done of vacuum and argon, kept at different time. After 3 cycles the sample is melted into a bead. It is melted at 80 amperes current, and is flipped 3 times.

(ii) **Making Ni_2MnGa**

To make Ni_2MnGa only Nickel bead was made using arc furnace and the bead was later done by first placing Mn then Ga and bead was placed

(a) Nickle bead formation

In order to prepare the Nickel bead first Nickle powder taken little in excess. The following table is about the weight taken before the process

Ni taken	Ni required
0.8123g	0.7276g

Table 2.5: weights

Three cycles were done of vacuum and argon, kept at different time. The following table gives us information of the bead formation process.

(b) Ni_2MnGa bead formation

The nickel bead, Manganese and gallium was placed in the copper earth.Three cycles were done of vacuum and argon, kept at different time. After 3 cycles the sample is melted into a bead. It is melted at 80 amperes current, and is flipped 3 times.

WEIGHT LOSS

After Nickel bead is prepared weight loss is calculated. Weight loss is calculated by Weight loss = [(initial wight – weight after melting)/ intial weight] x 100 Similarly, after the sample bead is prepared weight loss is checked

DIAMOND CUTTER

The Low-Speed Diamond Saw is a convenient preparation instrument in the laboratory and is designed for the precision cutting of a wide variety of materials. Samples can be cut with minimal damage and deformation. It has an adjustable motor speed used for various applications. Also, has removable catch tray for easy retrieval of cut sample. The blade of the diamond cutter is thoroughly dipped in diamond coolant. The coolant is made in the ratio 30:1 i.e. if 5 ml of coolant is used then 150 ml of water is mixed.

Figure 2.3: daimond cutter

(i) Cutting of HE Ni2MnGa

High Entropy Ni_2MnGa is extremely hard. Cutting the sample into two halves took 96 hours. And it was kept on speed 10. After cutting it into 2 halves, one half was taken and a slice of it was cut. Time taken to cut the slice was 48 hours. After we got the slice, it was then cut for resistivity. 3 parts of the slice were made.

(ii) Cutting of Ni_2MnGa

Ni_2MnGa is a very hard sample. Time taken to cut the sample into 2 halves is 6 hours. And it was kept on speed 10. After cutting it into 2 halves, one half was taken and a slice of it was cut. Time taken to cut the slice was 4 hours. After we got the slice, it was then cut for resistivity. 3 parts of the slice were made. Each cut taking 15 mins.

Figure 2.4: Cut sample

Annealing

After cutting the sample into 2 halves it was used for various characterization measurements such as XRD, four probe resistivity and DSc. Now one peice of the half is used to cut for resistivity peice and the other half is used for Xrd. This half which is being used for Xrd is crushed into fine powder using motar pestle. This fine powder is wrapped in tantalum foil. The resistivity peices, Dsc peice , the fine powder in tantalum foil and the remaining part of the half are sealed in vaccum using quartz ampoule. Lastly the sample is annealed at 750 degree celcius for 48 hours, followed by rapid ice-water quenching.

Figure 2.6: sample quenched

Figure 2.5: quartz ampoule in furnace

2.3 Characterization Techniques

2.3.1 XRD

X ray powder diffraction is a well-known rapid analytic technique basically used for the study of phase identification and crystal structure of material. X-ray diffraction is based on a crystalline sample and constructive interference of monochromatic X-rays. X-rays are basically generated by a cathode ray tube which is filtered to produce a monochromatic radiation, collimated to concentrate, and is directed towards the sample. Diffraction occurs only when the Bragg condition which states that, the X-rays reflected from two neighbouring planes of atoms must coherently interfere, is satisfied. Which is mainly the Braggs law (shown in the fig below) The Bragg's Equation is

$$2d \sin \theta = n\lambda \tag{2.16}$$

where,

n is an integer,

λ is the x ray wavelength,

d is the inter planar spacing,

θ is the diffraction angle

now what we learn from this law is that it tells us that the wavelength of electromagnetic radiation to the diffraction angle and the lattice spacing in a crystalline sample. These diffracted X-rays are then detected, processed and counted.An X-ray diffraction is carried out on the single crystals or polycrystalline (powder) samples. The XRD pattern is a plot of the intensity of diffracted rays versus Bragg angle which has information about the structure and composition of the basis. This information requires to be extracted from the pattern by doing a proper analysis procedure.

Now in powder method, the material to be studied is pounded to very fine powder and placed under a beam of monochromatic x-ray where each particle of the powder is a tiny crystal arranged in random direction with respect to incident beam. Because of random direction of the particles 19 Braggs reflection condition is satisfied for all the lattice planes. Hence by scanning the sample through a range of 2 angles, all the possible diffraction directions of the lattice should be acheived.

XRD WORKING

X-ray diffractometers mainly consist of three basic elements that is an X-ray tube, a sample holder, and a X-ray detector. X-rays are basically generated in a cathode ray tube by heating a filament to produce electrons which accelerats the electrons toward a target by applying a voltage, and bombards the target material with electrons. In vacuum, a high voltage

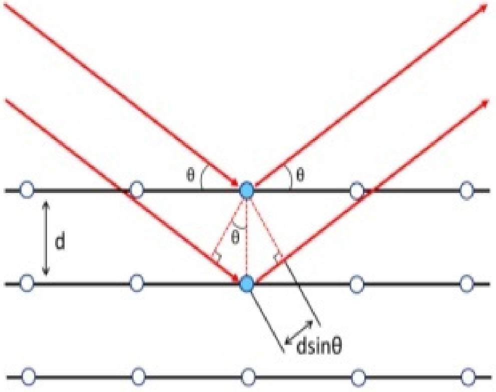


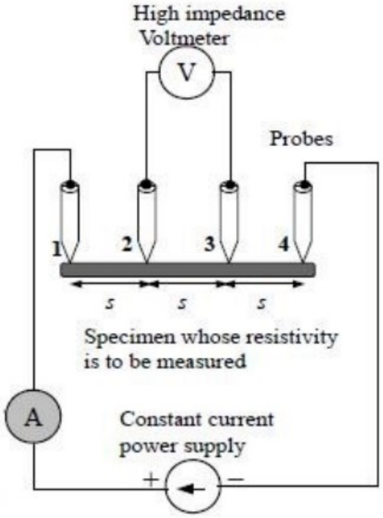
Figure 2.7: braggs law

between cathode and Anode is maintained.[3] Anode is water cooled block of copper containing desired target metal like Al, Mn, Mg etc, whereas the Cathode is filament of tungsten. on heating filament electrons are generated and accelerated towards a target by applying a voltage, and bombarding the target material with electrons. On hitting, the target electrons transfer their kinetic energy to its metallic surface and eject the core electrons generating hole. When higher orbit electrons jump into the hole generated in core orbits characteristic x-ray spectra are produced.There are other components in these spectra,[3] but the most prevalent ones are $K_\alpha$and K_β. Part of K_α is made up of $K_{\alpha 1} and$K$_{\alpha 2}$.K$_{\alpha 1}$ is twice as intense and has a slightly shorter wavelength than K$_\alpha$2. Cu, Fe, Mo, and Cr are the specific wavelengths that are indicative of the target substance. For the purpose of diffraction, monochromatic X-rays must be produced through filtering using foils or crystal monochrometers. Because $K_{\alpha 2}$ and $K_{\alpha 2}$ have similar wavelengths, a weighted average of the two is utilized. With CuK$_\alpha$ radiation = 1.5418Å, copper is the most often used target material for single-crystal diffraction. The sample is exposed to these collimated

X-rays. The intensity of the reflected X-rays is recorded while the sample and detector are rotated.[3]

2.3.2 Four probe resistivity

In the studies done in electrical properties it was noticed that four-probe setup is used to measure the resistivity of the alloy. The resistivity set up consist of sample holder which securely holds the sample in place, with four equidistant copper contacts , these contacts are often known as probes in which the outer two probes provide for passing current through the sample while the inner two are for measuring the constant current generator,[7] induced voltage, power supply and digital screen for measuring voltage and current across the probes.[2]



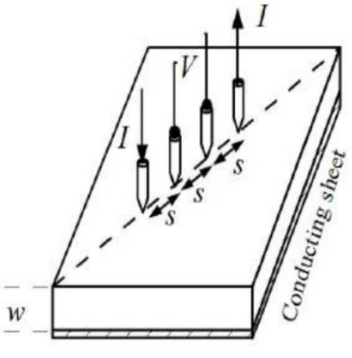


Figure 2.9: Four probe

Figure 2.8: Four probe resistivity

Using G-varnish the rectangular shape slab of sample was mounted on the sample surface. The G-varnish helps the sample to stick on the sample platform. Using fine layer of silver paint, four copper contacts were made on the sample surface.There is a manganin heater wound which is placed just below the sample holder this helps to control the temperature of the sample. Depending on the resistance of the sample at room temperature a constant current is passed through keithley 224 source meter. This device is connected to two outer probes of the sample. Also with the help of keithley 2128 nanovoltmeter poteintial drop is measured. Measured resistivity is converted into resistivity. The resistivity is calculated using,

$$Rho = R\frac{A}{L} \tag{2.17}$$

Where,

A is the area of cross section

L is the length of the sample.

2.3.3 Differential scanning calorimetry

Differential scanning calorimetry also known as DSC is a thermoanalytical technique in which it is used to measure the difference in the amount of heat released / absorbed by the sample and reference, mainly an empty pan crimped as function of temperature. Nearly same temperature is maintained throughout for both the reference and the sample. DSC is a very versatile technique used to study a huge range of materials. In DSC measurements, sample bearing weight of approximately 9 mg is carefully crimped and placed in an aluminium sample pan over which a lid is placed. Now with the help of tweezers the two pans are then placed into the sample and reference cell of DSC (DSC 60) .After which sample is mounted and from TA controller window,it is set to run a complete cycle.

Figure 2.10: DSC

Chapter 3

Results

3.1 XRay- Diffraction: XRD

Since we crushed the sample into fine powder after annealing there was stresses and strains in the sample due to which we got broader peaks and which were difficult to analyze. Below are the images of generated patterns show in fig 3.1 and In figure 3.2 is the the xrd data we received.

On the basis of data received a Ni_2MnGa we compared it with the generated phases to check which phase matches with it. After doing so, we learned that the cubic phase matches with it, This comparison is illustrated in the figure 3.3 which tells us that it matches with the cubic phase and has a space group of Fm−3m.

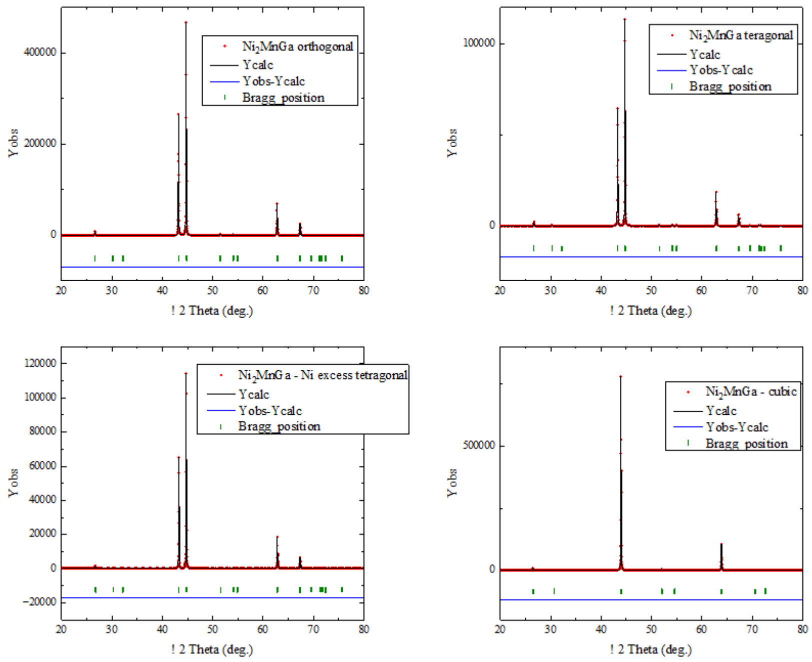


Figure 3.1: generated pattern for orthogonal , tetragonal , cubic , nickle excess

In High entropy Ni$_2$MnGa again generated phases where compared with XRD data received. Figure 3.4 is the XRD data recived. In the data provided an extra peak observed and we see that it approximately matches with the tetragonal phase which is illustrated in figure 3.5 and has a space group 14/mmm .

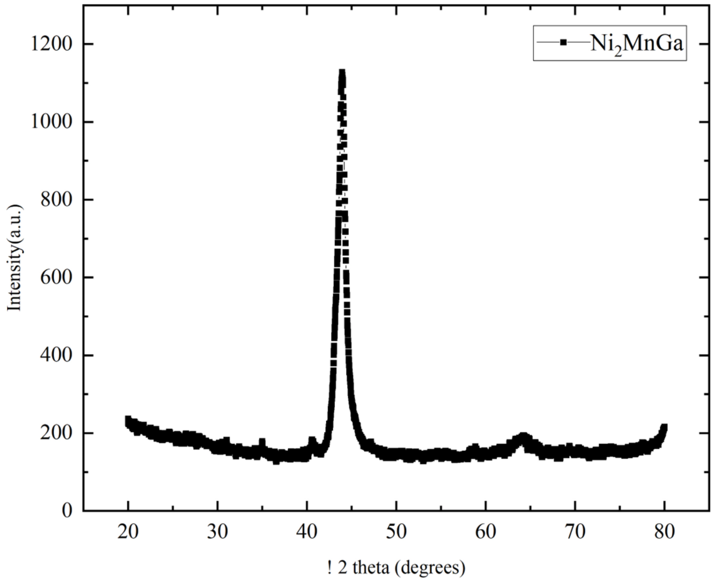


Figure 3.2: XRD data of Ni$_2$MnGa

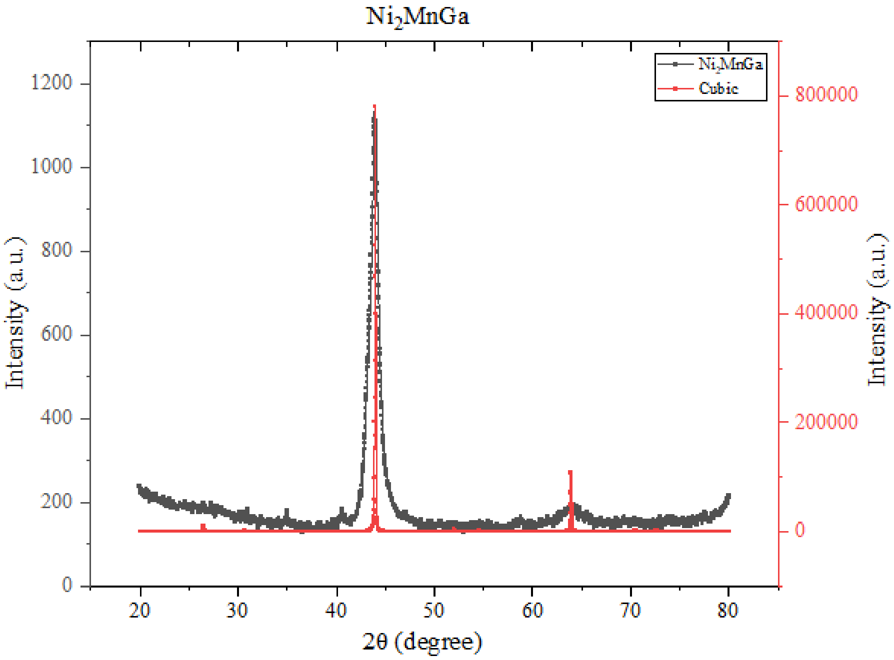


Figure 3.3: phase detection of Ni$_2$MnGa

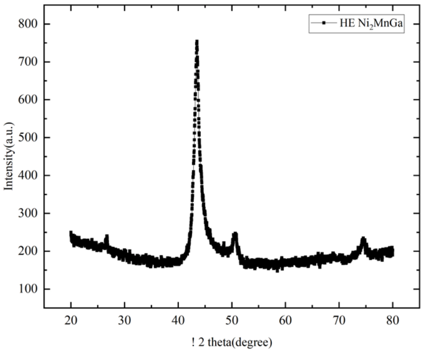


Figure 3.4: XRD data of HE Ni_2MnGa

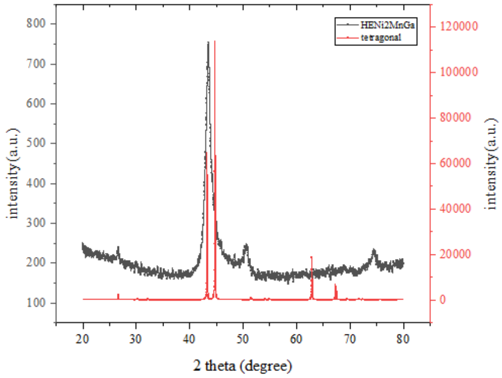


Figure 3.5: phase detection of Ni_2MnGa

3.2 Resistivity

To investigate a rough approximation of martensite transformation temperature, resistivity measurement was carried out on samples using four-probe resistivity setup. Below are the pictures of resistance plotted as the function of temperature for Ni_2MnGa and High entropy Ni_2MnGa in the range of temperature 50-450 K.The resistivity plot in Fig. 3.6 shows well a defined hysteresis loop , but this is reversible on heating and, thus, the alloy shows the shape memory effect and we also see a transformation temperatue around 270K and in High entropy Ni_2MnGa sample we can see that at lower temperarture in the first warming cycle the are some irregular jumps which become less as we run the cooling cycle and almost go as we conduct the warming cycle again. here also we cn see a transformation temperature around 270K hence both the results approximately match.

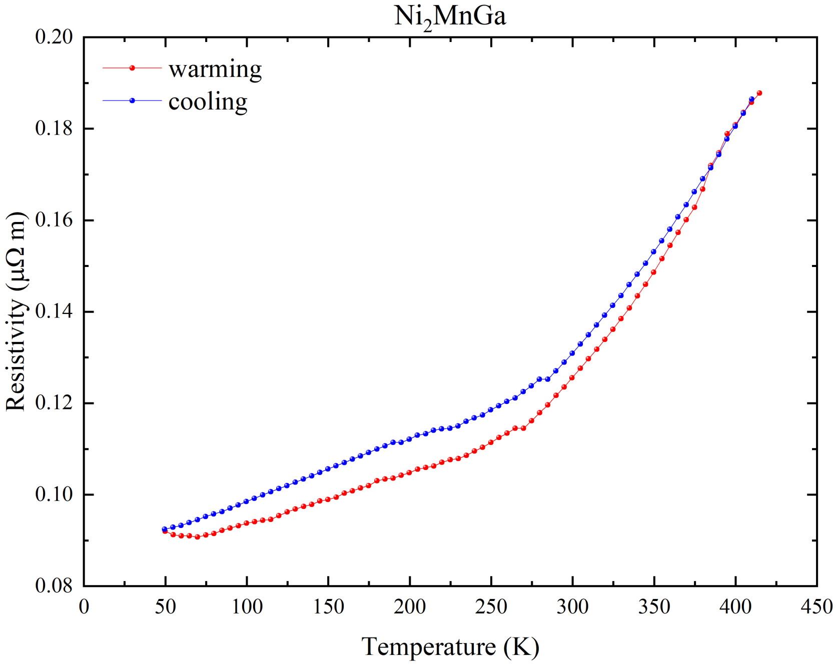


Figure 3.6: Resistance plotted as the function of temperature for Ni$_2$MnGa

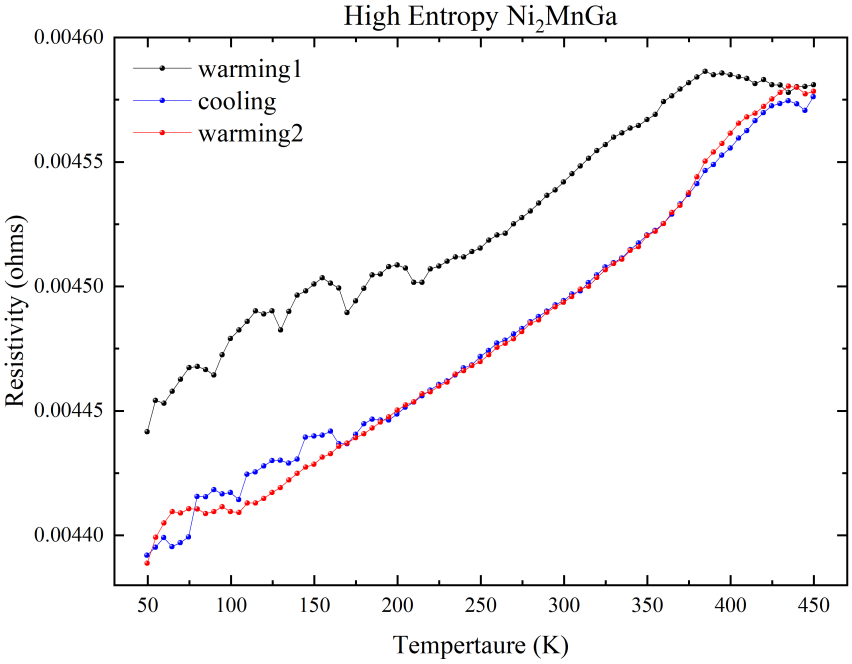


Figure 3.7: Resistance plotted as the function of temperature for HE Ni$_2$MnGa

To show the results more in details a comparison of both the samples is shown in above images in which we can see that around 270K there is a small disturbance which indicates that there is a transition at around that temperature

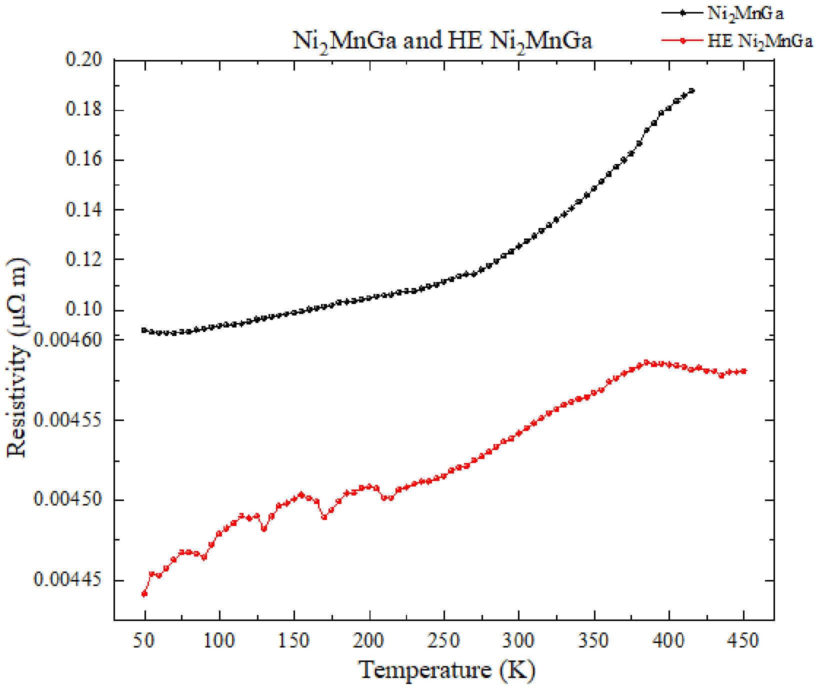


Figure 3.8: warming cycle of Ni$_2$MnGa and High entropy Ni$_2$MnGa

3.3 Differential Scanning Calorimetry

DSC helps us to know the transition temperature. This is the data for warming cycles of both the samples. From these data we see that the transition temperature is about 270K

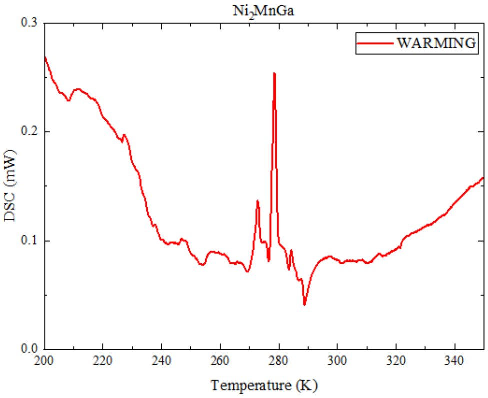


Figure 3.9: DSC data of HE Ni$_2$MnGa

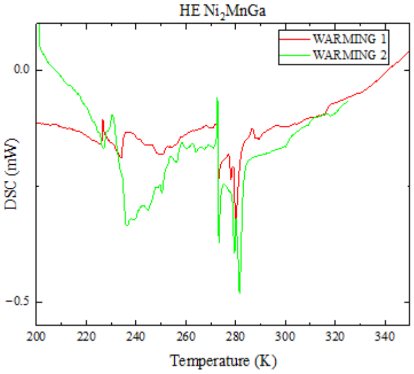


Figure 3.10: DSC data of HE Ni$_2$MnGa

3.4 conclusion

We prepared 2 alloys Ni_2MnGa and HE Ni_2MnGa and did various characterization techniques and in this we see that the sample of HE Ni_2MnGa there is an extra peak which is shown in the figure 3.11 and the resistivity plot shows there is a transition at around 270K which is shown in the figure 3.12

Hence both the results are verified

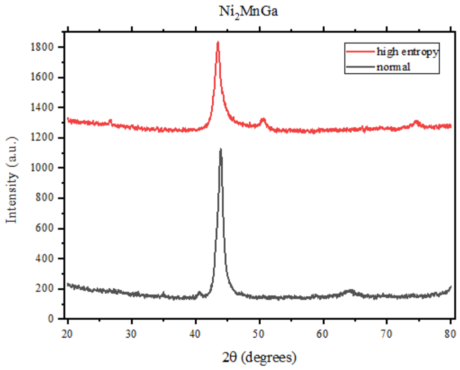


Figure 3.11: XRD

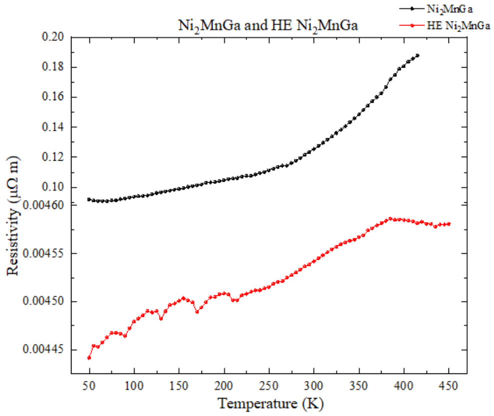


Figure 3.12: Resistivity

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[1]

[2]

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